

EXPERT KNOWLEDGE **TEST PROCEDURES** **OF ELASTOMER COMPONENTS**

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Integral Approach in Evaluating Elastomer Seal Damage Patterns – The Best Way to the Real Cause of Failure

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Every year, seal failures cause direct and indirect consequential costs that are certainly 2-3-digit million euros in economic damage, be it through machine downtime costs, recalls or environmental damage. Even worse than the direct damage can be the resulting image problems on the market for the affected companies. Therefore, it is of course very important to quickly identify the real cause of the failure in the event of damage in order to be able to eliminate the fault. For this reason, damage analysis is initially about much more than just finding the guilty party (which, by the way, is usually even afterwards not easy to determine). Therefore, damage analysis is of great importance in practice. However, the performance of a damage analysis is often reduced only to a material examination of the damage pattern in combination with a microscopic examination, possibly also with a scanning electron microscopic examination. However, these examinations alone can easily be misleading, as material inhomogeneities, foreign substances, traces of aging and slight manufacturing defects can almost always be found on failed rubber seals. The "art" then consists in interpreting the

existing traces correctly. This is only possible, however, with an integral approach, which means that all available information on the case of damage must be considered in the assessment of the damage picture so that the logic of the failure can be clearly identified. This article aims to show what information or knowledge is necessary or desirable and how it can be incorporated into the assessment of a damage pattern. Practical examples of damage cases will then be used to illustrate this.

The 5 Components of a Reliable Damage Analysis

As with many complex tasks, a systematic approach also helps in damage analysis. The system developed in the O-Ring Prüflabor Richter from numerous (over 1800) investigations consists of five steps which are discussed below:

1. Identification of the Sample
2. Obtain Application Information
3. Documentation of the Damage Pattern with Evaluation and/or Classification
4. Ensuring that the Conclusions Drawn from Step 3 are Validated
5. Generating a Report with Possible Corrective Actions to Eliminate the Error

Step 1: Identification of the Sample

A damage analysis can only begin when the damage pattern is actually present. Quick shots based on pictures and application information alone are not sufficient for a reliable analysis and can mislead even experienced specialists. The first step in examining the damage pattern is to determine whether the material complies with the specifications. As the simplest identity check, the hardness and density test can provide first clarity (compliance with the order specification or the supplier information). In addition, an FTIR analysis can be used if necessary to ensure the correct polymer (e.g. FKM or EPDM). In approx. 1-2% of the cases of damage, the damage analysis is therefore complete if it turns out that the wrong material was used. It is recommended to examine a currently used series pattern in comparison to the damage pattern in parallel to the examination of the damage pattern. This quickly shows the changes that have taken place, and also allows the examination of the quality of any defects assumed to be the cause of the damage. The identification of the sample includes not only the material but also the dimensions of the sample. Here, the essential functional dimensions of the damage pattern should be compared with those of the standard pattern: in the case of O-rings, for example, the cord thickness and the inner diameter; in the case of lip seals or radial shaft seals, the inner and outer diameters.

The first step is to prove that the correct material and dimensions are present and that any material and dimensional changes have been documented.

Step 2: Obtain Application Information

The second step is to understand the application and the whole history of the failure. First and foremost, this involves critically questioning whether it was established beyond doubt that the gasket under investigation had actually been identified as the cause of the leakage. Then it is very important for further evaluation to know when the leaks occurred. Directly or promptly

(<100-1000 operating hours) after start-up, or only after a considerable period of operation (>1000h). In the case of failures after short operating times, the initial focus is on assembly or manufacturing defects as a possible cause, while in the case of longer operating times, more attention is paid to impermissible thermal, chemical or physical stresses. In addition, the type of leakage should be known: is there a coarse fluid leakage or "only" a droplet or sweat leakage? Is it "only" a matter of increased gas leakage rates, which have been determined by leakage detectors, or do leakages especially occur under certain conditions, for example at low temperatures? It is also important to know whether the failures occur with different users or only with one user or whether the failure is just a single case. If there are several failures, can a logic be recognized, for example from a certain production time? Or is there a certain production batch affected by it? Or do failures only occur regionally or seasonally? Then it is important to know whether the leaking component has just been manufactured for a short time or whether this component has been working without problems for many years. It can also be of help if the client of the damage analysis already has a concrete suspicion. If, for example, operating mediums or application conditions at the customer have changed, or if there is a new supplier for the gasket, or if there is a new injection tool for the installation space of the gasket. Another important prerequisite for effective damage analysis is to have understood the application. This means having understood the functionality of the seal, diaphragm or elastomer component. Only after understanding why the application with the component has worked so far can one understand why it no longer works. This provides an important conclusion as to whether the cause of failure is more to be found in the seal or in the design of the installation space. This leads to a further essential element of this second step of the damage analysis, the evaluation of the installation space. In order to be able to evaluate this, corresponding information should of course be available, but if necessary the installation space can also be evaluated using the failed component. In the end, one should also allow the question: how reliable is the information given? What uncertainties exist with regard to the operating conditions mentioned, are there any indications that certain information is being withheld? This can become important at the end of the investigation, if one thinks to have understood the damage picture and therefore the origin of the damage. If this assumption then contradicts the information given on the operating conditions, then either the assumption made on the occurrence of the damage is wrong and the logic of the failure has not yet been recognized, or the information given on the operating conditions is incorrect.

The intermediate result of the second step should be to recognize whether further investigations should primarily concentrate on the seal or the installation space. Furthermore, it can be discerned whether the client actually wants an open-ended damage analysis, or whether an independent expert should put the client's findings on paper. Fortunately, the latter is rather rare in the day-to-day operations of the O-Ring Prüflabor Richter. Generally, however, the results of the next step, the evaluation of the damage pattern, are too compelling to lead to a serious conflict with the client even in these cases.

Step 3: Documentation and Evaluation of the Damage Pattern

This third step is, practically, the most important part of the damage analysis. Hereby it is now a matter of recognizing first of all the traces of the stress on the gasket and then evaluating them correctly. A good microscope can therefore considerably increase the reliability and effectiveness of a damage analysis. In the O-Ring Prüflabor Richter we have been working

with a digital microscope (Keyence VHX 500) for several years and in 2014 we updated to the latest state of the art (Keyence VHX 5000). Usually we work in the magnification range 20-200x, while in exceptional cases we use a lens with 250-2500x magnification. Using a panorama image function (VHX 5000) or with an additional lens (VHX 500), it is also possible to take pictures with a lower magnification. At first the undestroyed sample is examined for abnormalities, which is then sometimes also documented three-dimensionally with high magnification and also measured. The specimens are then cut open to detect permanent deformations or crack formations, internal cracks, production inhomogeneities or bubble formation. Fracture surfaces are of course also documented and evaluated. A 3D representation is a considerable help here.

In the subdivision or classification of the damage mechanisms, the subdivision into 4 classes of causes has been useful, see also **Figure 1**:

- **1st cause** = medium-damage caused either by inappropriate swelling (in exceptional cases also severe shrinkage) or by chemical impact, meaning loss of rubber elasticity, cracking and/or severe permanent deformation.
- **2nd cause** = temperature/Aging - damage can be caused by a strong overheating, which went far beyond the permissible continuous temperature and therefore led to a surface damage of the seal, which is usually shown in crack formation. The damage could also result in embrittlement and permanent deformation within the polymer-typical temperature limits due to excessively long operating times (polymer does not suit the application, e.g. NBR for hot water applications). The use of a poor state of the art in formulation design (e.g. Sulphur cross-linked EPDM instead of peroxide cross-linked EPDM) can also be a cause of damage. Included in this group are also related damage mechanisms, which ultimately lead to premature failure through damage to the network structure of the material. These are different forms of aging, for example due to static deformation and ozone (usually on pre-assembled NBR O-rings) or due to the presence of heavy metal ions (e.g. on EPDM O-rings in hot water systems).
- **3rd cause** = inadmissible physical stresses - this includes all failure causes that can explain a failure without having caused changes in the network structure of the material and without the failure being the result of a manufacturing fault. The addition "mechanical" should emphasize that this also includes assembly damage, or other typical causes are sharp-edged installation spaces, too little or too high compression, gap extrusion, abrasion or explosive decompression or explosive overheating.
- **4th cause** = manufacturing defect - this includes defects that are directly attributable to the manufacturing process and also clearly represent an impermissible deviation from the target condition. The most common defects in O-rings here are cracks or radial flow lines, a preliminary stage to cracks. Also, with other seals, manufacturing defects can lead to cracks even under low mechanical stress, for example if a superimposed mixture has been processed. Further possible defects are demolding cracks, which can be explained by the high tear sensitivity of elastomers at high temperatures which can occur when the elastomer parts are demolded.

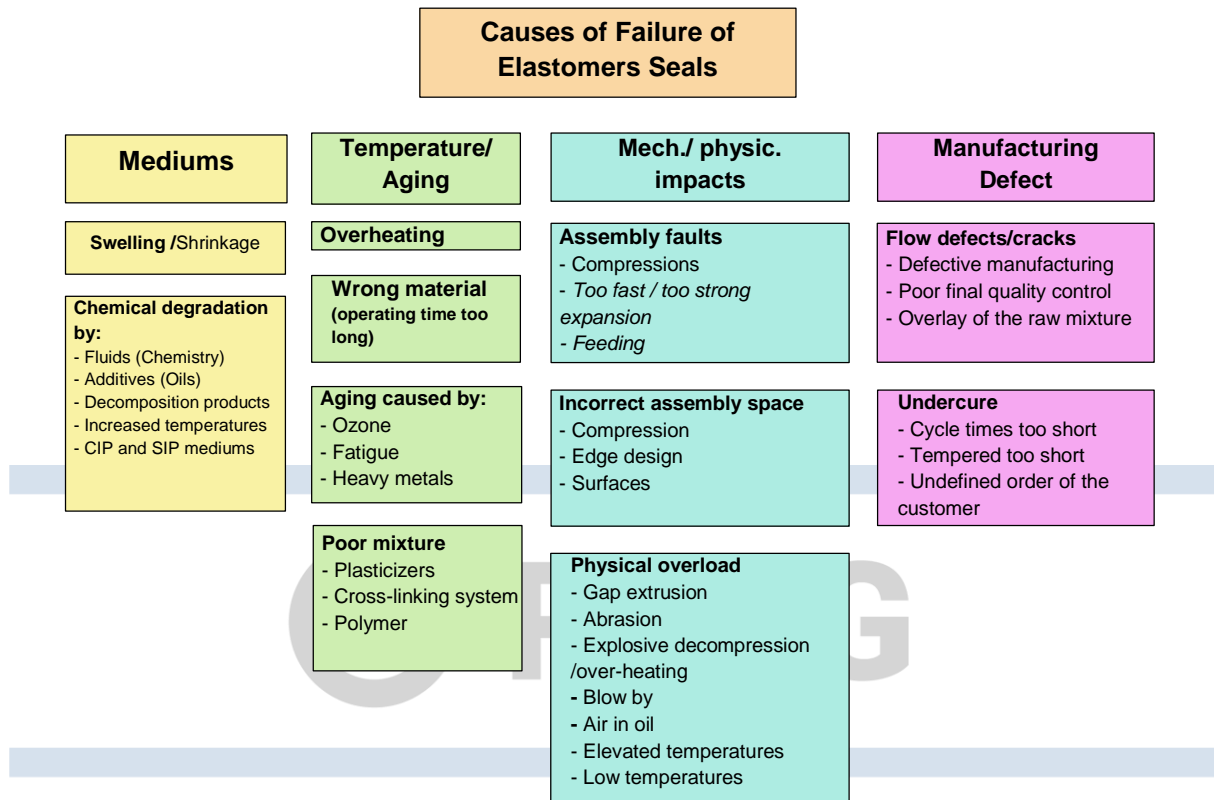


Figure 1: Classification of the failure causes of elastomer seals

When classifying the defect pattern, the exclusion principle worked well by first trying to exclude impermissible chemical and thermal causes (loss of rubber elasticity, cracks, strong permanent deformation or strong swelling/strong shrinkage). If these can be excluded, an attempt is made to exclude manufacturing defects. An important criterion for manufacturing defects as a cause of failure is the short operating time until failure (<100-1000h). If this can also be ruled out, then only impermissible physical effects on the seal remain as the cause. An important result of this third step is the detection of damages, which can explain a leakage (if not, one has to concentrate on the installation space), and the assignment of the fault to a main group according to **Figure 1**. After this evaluation an assumption for a damage mechanism should be found, which means the derived damage hypothesis should not contradict existing information, see step 2. This may lead to further specific consultation with the client, which may support the damage hypothesis. A further important result of this described evaluation of the damage pattern should also be to determine further analytical tests (TGA, FTIR, EDX, DSC, GC-MS) in order to find indications of diffused mediums, modified formulations, the presence of heavy metal ions or undercure. This means that the benefit of analytics consists primarily in supporting or invalidating the damage hypothesis resulting from the evaluation of the damage pattern. The use of complicated analytics without this decisive step can cause more confusion than clarity.

Step 4: Validation of the Assumptions

If a possible explanation for the cause of the damage has now been found in step 3, this assumption must be supported as well as possible. This could include to ask clarifying

questions to the user. For example, in the case of mechanical damage to a symmetrical hydraulic seal (which is a physical damage mechanism), the question of whether the damage to the sealing gap occurs on the low-pressure side is important. If this is the case, gap extrusion would be an explanation, while if it occurs on the pressure side, air would be a possible cause. If there are indications of assembly-related errors, the assembly process at the customer should of course be questioned in detail.

Using analytics for instance can clearly prove if there is a chemical degradation, as for example on an EPDM membrane, which was supposedly only used in water, whether traces of a disinfectant (chlorine) can be found on the damaged surface (e.g. via an EDX analysis). A comparison by means of thermogravimetric analysis TGA between the new reference part and the failed seal can prove that an extraction of plasticizers has taken place. In the case of NBR seals, the acrylonitrile content of the formulation could be compared with the initial sample via the DSC cold standard value. If this is unchanged and therefore also the swelling resistance, swelling can only be explained by modified oils.

The 4th step in the damage analysis therefore serves to confirm the assumptions made in step 3. Due to the further development of analytics, the possibilities today are much greater than they were 10-20 years ago. Wherever possible, these options should be used to determine with the greatest possible certainty the cause of the seal failure (root cause), such as contact mediums of the seal, presence of heavy metals or modified seal formulations.

Step 5: Report Writing / Proposal of Remedial Measures

The report production represents the conclusion of the damage analysis. Here, however, it is not only a matter of naming the assumed cause of the damage, but to present a complete logical explanation of the failure, so that it is also comprehensible to readers who are not involved or particularly proficient. Only then can it be expected that the client will also accept the proposed damage causes and that the necessary remedial measures will be implemented. For this reason, it is necessary to document and comment well on the error patterns found. The objective results from the microscopic and material examinations must be well presented and evaluated. If there are different possible evaluations of the results, it should also be dealt with in the report. Often it is also helpful to explain which damage mechanisms can be excluded and why. If the expert preparing the damage analysis is left with uncertainties in the evaluation of the damage pattern and therefore in the naming of the assumed main cause, it should also be reflected in the report.

If, in the final analysis, it can be assumed that the real cause of the damage has been identified, the report should of course address the possibilities for remedial action.

Practical Examples

The following examples are listed in tabular form to illustrate the procedure for real cases.

Example 1

| | |
|------------------------|---|
| 1. Step-Identification | HNBR O-ring, hardness 68 Shore A, density 1.16g/cm ³ , FTIR-Analysis: HNBR |
|------------------------|---|

| | |
|-------------------------------------|---|
| 2. User Information | 1500 h, T>100°C, Engine oil, preload ring for rotating sliding element |
| 3. Evaluation of the Damage Pattern | cracks, conspicuous fracture surface, see pictures 2 and 3, cracks appear inside, explosive overheating |
| 4. Assurance of the Assumptions | recommendation: oil analysis for blow-by condensates |
| 5. Remedial Measures | avoidance of cyclic loading, check of start-up process, inspection of oil |



Figure 2: Example 1 - Cracks on the outer diameter



Figure 3: Example 1 - Fracture surface

Example 2

| | |
|------------------------|---|
| 1. Step-Identification | NBR O-rings, bonded, 2 different dimensions, New: Hardness, 75 IRHD, density 1.22g/cm ³ Fail-safe part: 81/82 IRHD, 1.24 g/cm ³ No significant dimensional changes |
| 2. User Information | Temperature < 120°C (1st O-Ring) < 50°C (2nd O-ring) Static sealing, crack at the joint after approx. 1000h at about the same time at 3 O-rings |

| | |
|-------------------------------------|---|
| 3. Evaluation of the Damage Pattern | Crack at the adhesive joint, see Figs. 4 and 5, with supposedly little thermally loaded O-rings near the joint crack, see Fig. 6 , assumption of thermal damage, no quality problem with the joint |
| 4. Assurance of the Assumptions | Tensile test on new reference sample, elongation at break >100%, tensile tests at customer's site also ok. |
| 5. Remedial Measures | Find cause for overheating, better use endless vulcanized O-rings with better temperature resistance |



Figure 4: Example 2 - Torn joint

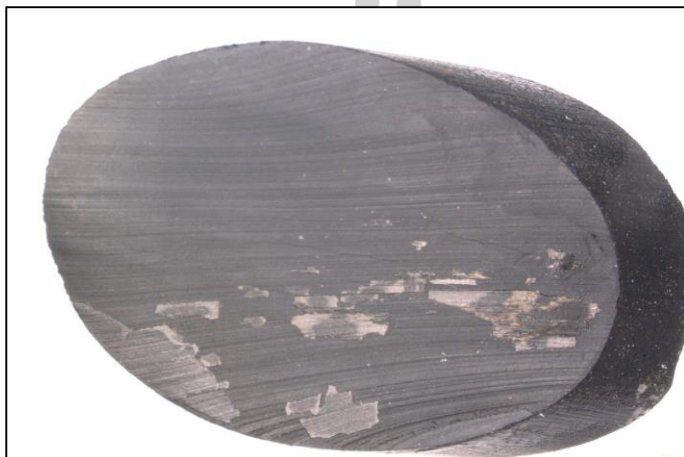


Figure 5: Adhesive residues (acrylate adhesive) at the butt joint

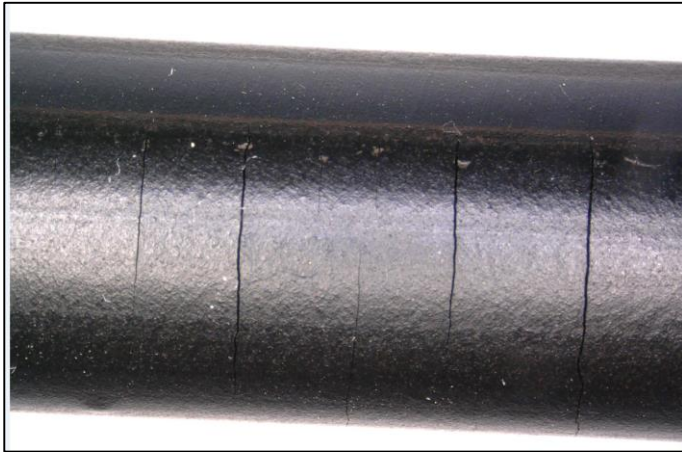


Figure 6: Cracks during bending at the O-ring with the supposedly lower thermal load

Example 3

| | |
|-------------------------------------|--|
| 1. Step-Identification | NBR fuel hose, hardness 72 IRHD, density 1.36 g/cm ³ |
| 2. User Information | NBR fuel hose with a braiding of galvanised steel wire, leaks after 4-5 months. Pumped medium is Diesel |
| 3. Evaluation of the Damage Pattern | Directed cracks, see Fig. 7 , the profile section shows that the cracks are formed on the outside, which means they are not caused by the pumped medium on the inside, see Fig. 8 . The damage pattern is typical for ozone cracks |
| 4. Assurance of the Assumptions | Ozone test 48h/23°C/20% elongation 50pphm Ozone on new reference part, result strong cracks, see Fig. 9 |
| 5. Remedial Measures | Conversion to new formulation (and new supplier), repetition of ozone test, no cracks in results, see Fig. 10 |

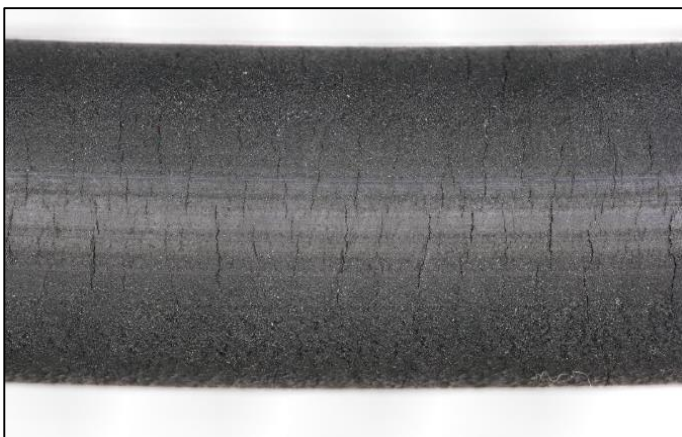


Figure 7: Example 3 - Cracked NBR fuel hose

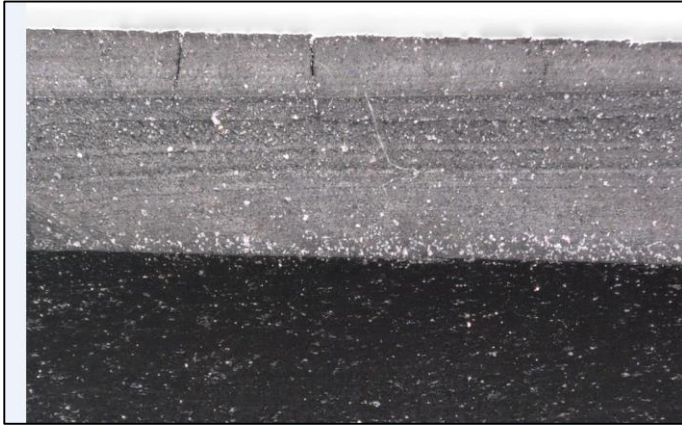


Figure 8: Example 3 - Profile section of the hose wall

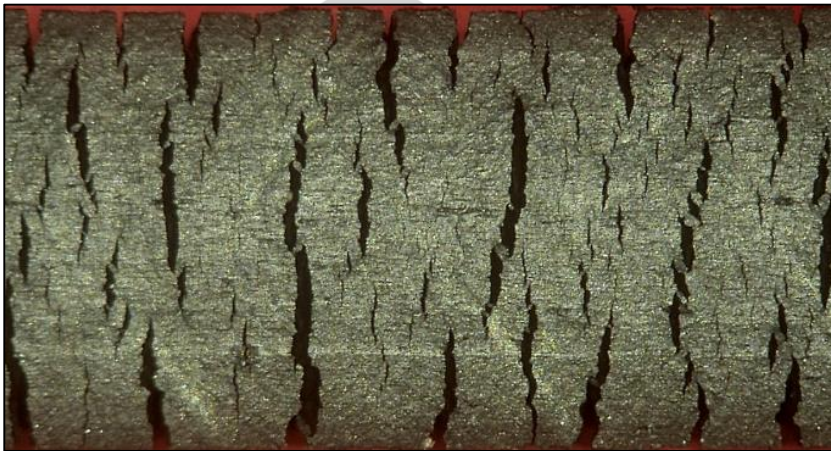


Figure 9: Example 3 - Current series sample after ozone test



Figure 10: Example 3 - New material (new supplier) after ozone test